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PATENT SPECIFICATION

817,622

Inventor:—WILLIAM EDYVEAN.



Date of filing Complete Specification : Jan. 15, 1958.

Application Date : March 4, 1957. No. 7125/57.

Complete Specification Published : Aug. 6, 1959.

Index at Acceptance :—Classes 1(1), A3B(1:2X); and 2(3), C3A14A(1A:1B:7B).

International Classification :—B01j. C07c.

COMPLETE SPECIFICATION.

ERRATUM

SPECIFICATION NO. 817,622

Page 3, line 84, for "100 hours" read "1000 hours".

THE PATENT OFFICE,
9th November, 1959

DB 28397/1(9)/3866 150 11/59 R

... large number of catalysts are suitable for use in the production of ketones by the continuous catalytic dehydrogenation of secondary alcohols, including a wide variety of metals, metal oxides and mixtures thereof. For example ketones such as acetone, methyl ethyl ketone and diethyl ketone may be prepared from the corresponding secondary alcohols by dehydrogenation in the presence, as catalyst, of a metal such as copper or zinc or an alloy thereof such as brass. Oxides of many metals or mixtures of metals and oxides of the same or different metals, as for example oxides or mixtures of oxides supported on copper, zinc or brass are also suitable. If desired the oxide catalysts can be supported on inert materials such as pumice, majolica or porcelain which take no significant part in the reaction and act merely as mechanical supports for the catalytically active material. Alloys of copper and zinc may advantageously be used, particularly those which have been pickled in mineral acid or for example acetic acid to produce a granular catalyst having a catalytically active outer layer. Many of these dehydrogenation catalysts have already been proposed for use

between 380 and 420° C. initially, rising to say 480 to 500° C. as the reaction proceeds. The reaction may be effected under atmospheric or superatmospheric pressures such as up to about 5 to 10 atmospheres. Such tubular reactors may take a variety of forms, and may include the case in which some of the tubes contain no catalyst and serve primarily to vaporise the liquid alcoholic feed. It will be appreciated that other forms of reactor may also be used, as for example a single reactor containing either a fixed or a fluidised bed of catalyst.

Although the process is carried out in the vapour phase it is conveniently started up by passing the liquid feed through the whole of the reactor which is gradually heated to reaction temperature. In the case, for example, of a tubular reactor of the kind referred to above, which is heated by hot gases, the last row of tubes will be the first to reach working temperature and consequently vaporisation of the alcoholic feed will occur in those tubes first. As the plant heats up the liquid/vapour interface moves back through the tubes until, under reaction conditions, vaporisation will be taking place in the first row of tubes and vapour phase

[Price ...]

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International Classification :—B01J. C07a.

COMPLETE SPECIFICATION.

Improvements in or relating to Catalytic Processes for Preparing Ketones from Secondary Alcohols.

We, "SHELL" RESEARCH LIMITED, a British Company, of St. Helen's Court, Great St. Helen's, London, E.C.3, do hereby declare the invention, for which we pray
5 that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the preparation of ketones from secondary alcohols, by a process in which dehydrogenation is effected catalytically.

A large number of catalysts are suitable for use in the production of ketones by the continuous catalytic dehydrogenation of secondary alcohols, including a wide variety of metals, metal oxides and mixtures thereof. For example ketones such as acetone, methyl ethyl ketone and diethyl ketone may be prepared from the corresponding secondary alcohols by dehydrogenation in the presence, as catalyst, of a metal such as copper or zinc or an alloy thereof such as brass. Oxides of many metals or mixtures of metals and oxides of the same or different metals, as for example oxides or mixtures of oxides supported on copper, zinc or brass are also suitable. If desired the oxide catalysts can be supported on inert materials such as pumice, majolica or porcelain which take no significant part in the reaction and act merely as mechanical supports for the catalytically active material. Alloys of copper and zinc may advantageously be used, particularly those which have been pickled in mineral acid or for example acetic acid to produce a granular catalyst having a catalytically active outer layer. Many of these dehydrogenation catalysts have already been proposed for use
40

in ketone production, and many proposals have also been made for improving the initial activity of such catalysts.

The preparation of ketones by catalytic dehydrogenation of secondary alcohols may be carried out by passing the appropriate secondary alcohol in vapour form through a reactor consisting of, for example, a bank of tubes containing the dehydrogenation catalyst, the tubes being heated to a reaction temperature lying between about 300 and 500° C.: thus the temperature may be between 380 and 420° C. initially, rising to say 480 to 500° C. as the reaction proceeds. The reaction may be effected under atmospheric or superatmospheric pressures such as up to about 5 to 10 atmospheres. Such tubular reactors may take a variety of forms, and may include the case in which some of the tubes contain no catalyst and serve primarily to vaporise the liquid alcoholic feed. It will be appreciated that other forms of reactor may also be used, as for example a single reactor containing either a fixed or a fluidised bed of catalyst.

Although the process is carried out in the vapour phase it is conveniently started up by passing the liquid feed through the whole of the reactor which is gradually heated to reaction temperature. In the case, for example, of a tubular reactor of the kind referred to above, which is heated by hot gases, the last row of tubes will be the first to reach working temperature and consequently vaporisation of the alcoholic feed will occur in those tubes first. As the plant heats up the liquid/vapour interface moves back through the tubes until, under reaction conditions, vaporisation will be taking place in the first row of tubes and vapour phase
80

[Price

ketone conversion will be occurring in all the catalyst-containing tubes, though mainly in the later row or rows of tubes. It will be appreciated that ketone production occurs gradually as warm-up proceeds and as the catalyst reaches working temperatures. Such a warm-up period, which includes the initial condition in which liquid alcoholic feed contacts substantially the whole of the catalyst, will hereinafter be referred to as the "initial stages" of the process.

As in the case of many catalytic reactions carried out on a technical scale, the activity of the dehydrogenation catalyst used in aliphatic ketone production tends to decline during the course of the process and it is usual in practice gradually to raise the reaction temperature as the reaction proceeds in order to compensate for this decline in activity. There is however a limit to the extent to which the reaction temperature can be raised and consequently it has hitherto been necessary to stop the reaction when the maximum working temperature has been reached and to remove the catalyst and either to regenerate it or to replace it with fresh catalyst altogether. Thus, for example, in processes for the preparation of acetone from isopropyl alcohol using a brass spelter catalyst it has been found necessary to remove the catalyst at intervals and to pickle it in mineral acid in order to remove surface impurities and to expose a fresh catalytically active surface. It is particularly desirable to improve the activity of the catalyst so as to increase the conversion obtainable therewith and since the removal and regeneration of the catalyst also adds to the cost of ketone production it is also desirable to reduce this expenditure as far as possible by improving the nature of the catalyst or the regeneration treatment so as to prolong the period before regeneration is required.

It has now been found possible to activate catalysts used in the dehydrogenation step of ketone production *in situ*.

Accordingly the present invention provides a continuous vapour phase catalytic dehydrogenation process for the preparation of a ketone from a secondary alcohol in which the catalyst is activated by contacting it with the secondary alcohol in a liquid state and containing at least one metal compound which will decompose to form a catalytically active deposit on the catalyst.

The compound or compounds contained in the secondary alcohol during catalyst activation in accordance with the present invention is preferably a salt or salts (advantageously an organic acid salt or salts, for example a salt or salts of acetic acid) of the catalyst metal (or of one or more of the catalyst metals if there is more than one catalyst metal) which will decompose subsequently to deposit a catalytically active

compound of the metal or metals, usually an oxide, on the catalyst, thus increasing the activity of said catalyst in use.

The catalyst is usually activated in accordance with the present invention at intervals during operation of the plant, for example at periodic intervals of between 500 and 1000 hours. A significant improvement in catalyst activity is thereby obtained, the catalyst being activated *in situ* in the reactor in such a manner that there is no possibility of the metal compound or compounds deposited on the surface of the catalyst being removed by the liquid starting material during the initial stages of the process. Also there is no danger of the catalytically active material deposited on the surface of the catalyst being removed accidentally in the handling and charging operations which would otherwise be required if the catalyst was activated in a separate operation effected outside the reactor, quite apart from the advantage the activation *in situ* is more convenient and less costly than activation in such a separate operation. Moreover, it has been found that a greater amount of catalytically active material can be deposited on the catalyst when the treatment is carried out *in situ* in the reactor in accordance with the present invention and that as a result of the treatment the activity of the catalyst—as represented by the degree of conversion which can be achieved therewith—is significantly enhanced.

However it is also within the scope of the present invention to effect catalyst regeneration in a catalytic process as defined above by interrupting the process, allowing the plant to cool down to below the vaporisation temperature of the liquid feed (such temperature being above the ambient temperature), and thereafter treating the catalyst with said metal compound or compounds in the manner defined above during the initial stages of the subsequent run.

The total amount of said compound or compounds added, either in the initial activation of the catalyst or in a subsequent regeneration treatment (e.g. in the manner just defined), may be between 1 and 10, e.g. about 6 to 7 pounds per ton of catalyst in use.

When a metal salt or a plurality of metal salts is employed it is convenient to add the salt or salts to the secondary alcohol in aqueous solution, although addition in an alcohol-miscible non-aqueous medium is also possible providing appropriate metal compounds are used.

The process of the present invention is particularly suitable for the production of the lower aliphatic ketones from secondary alcohols containing up to about 4 carbon atoms in the molecule. The alcohol feed may be substantially anhydrous or it may

be in admixture with water, e.g. as an azeotrope or constant boiling mixture. Such constant boiling mixtures are usually formed in the industrial preparation of the alcohols by hydration of the corresponding olefines and consequently can conveniently be used as feedstocks for ketone production.

In general the dehydrogenation catalysts which can be used in the preparation of aliphatic ketones from aliphatic secondary alcohols in accordance with the present invention contain one or more metals such as copper, silver, chromium, manganese, nickel, tungsten, cobalt, iron, cadmium, uranium, tin and zinc, either as such or in the form of their oxides or hydroxides (which may be formed by decomposition of the metal salts of organic or inorganic acids), and these catalysts may be used alone or supported on inert materials such as kieselguhr, pumice or majolica. The use of support materials such as activated alumina having dehydrating activity should be avoided otherwise dehydration of the alcohol feed occurs to give appreciable quantities of olefin as an unwanted by-product. A particularly suitable support for a metal oxide catalyst such as copper oxide is porcelain.

Whilst the present invention in its broadest aspect is not concerned with the particular catalyst employed in the dehydrogenation reaction or the method of preparing the catalyst, it has been found to be particularly advantageous when the catalyst contains the metals copper and/or zinc or an oxide or oxides thereof, which oxides may be supported on the metals or on inert supports such as porcelain, for example, in the form of beads or rings. An alloy of copper and zinc such as brass, for example, in the form of brass spelter which has been pickled in acid to expose a catalytically active surface, may also be used as the catalyst and/or support.

Thus according to a preferred feature of the present invention catalyst activation in a continuous process for preparing an aliphatic ketone from the corresponding secondary alcohol by dehydrogenation thereof in the presence of a dehydrogenation catalyst which contains the metals copper and/or zinc or an oxide or oxides thereof, is effected by adding a zinc salt and/or copper salt to the liquid alcoholic feed supplied in the catalyst activation stages of the process.

The salt is preferably an organic acid salt, for example, zinc acetate which has been found to be particularly effective for activating a brass spelter catalyst used for example in the production of acetone from isopropyl alcohol and which has been pickled in mineral acid prior to being charged to the reactor. However, other organic acid salts of zinc and/or copper

may be used, the salts being deposited on the catalyst during the initial stages of the process and thereafter decomposing to give the oxide.

The process of the present invention may be illustrated by the following Example:—

EXAMPLE.

Acetone was prepared from isopropanol using the isopropanol/water constant boiling mixture as the feedstock. This feedstock was passed in the vapour phase over a brass spelter catalyst in a tubular type reactor having narrow tubes of less than 6 inches diameter. The catalyst, which had been previously pickled in a mineral acid bath, was maintained at about 400° C. during the early part of the process, the temperature being raised gradually to a maximum of about 490 to 500° C. as the reaction proceeded.

Comparative runs were carried out (a) without any *in situ* catalyst treatment and (b) with the following treatment of the catalyst:—during the initial stages of the process 0.5 gallon of 10% (by weight) aqueous zinc acetate was added to the liquid feed at hourly intervals for a period of 8 hours.

Run (b) continued for over 100 hours with not less than a 70 mol per cent conversion. In run (a), that is, without *in situ* catalyst treatment, the process continued for only about 700 hours before the conversion fell below 70 mol per cent.

WHAT WE CLAIM IS:—

1. A continuous vapour phase catalytic dehydrogenation process for the preparation of a ketone from a secondary alcohol in which the catalyst is activated by contacting it with the secondary alcohol in a liquid state and containing at least one metal compound which will decompose to form a catalytically active deposit on the catalyst.

2. A process as claimed in Claim 1, in which the metal compound is a salt of an organic acid.

3. A process as claimed in the preceding claim in which the organic acid is acetic acid.

4. A process as claimed in any one of the preceding claims in which the catalyst contains zinc.

5. A process as claimed in the preceding claim in which the catalyst is a copper-zinc alloy.

6. A process as claimed in any one of the preceding claims, in which the metal compound contained in the secondary alcohol is a compound of a metal of the catalyst.

7. A process as claimed in any one of the preceding claims in which the metal

compound contained in the secondary alcohol is a zinc compound.

8. A process as claimed in any one of the preceding claims, in which the weight of the metal compound or compounds contained in the secondary alcohol is between 1 and 10 pounds per ton of catalyst in use.

9. A process as claimed in the preceding claim, in which the weight of the metal compound or compounds contained in the secondary alcohol is between 6 and 7 pounds per ton of catalyst in use.

10. Processes for the preparation of

ketones as claimed in Claim 1 substantially as described herein with special reference to the Example.

11. Ketones whenever prepared by a process as claimed in any one of the preceding claims.

DOWNES & ROBBINS,
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Agents for the Applicants.

PROVISIONAL SPECIFICATION.

Improvements in or relating to Catalytic Processes for Preparing Ketones from Secondary Alcohols.

20 We, "SHELL" RESEARCH LIMITED, a British Company, of St. Helen's Court, Great St. Helen's, London, E.C.3, do hereby declare this invention to be described in the following statement:—

25 The present invention relates to the preparation of oxygen-containing organic compounds, such for example as ketones and alcohols, by processes in which dehydrogenation or hydrogenation reactions are effected catalytically. The present invention is concerned particularly, though in no way exclusively, with processes for preparing ketones by catalytic dehydrogenation of the corresponding secondary alcohols and with the activation of catalysts for use therein and in order to simplify description the invention will be described in connection with such processes. It will be understood, however, that the present invention is in no way limited to catalytic dehydrogenation reactions and to the activation of catalysts for use herein but may be applied for example to catalytic hydrogenation reactions, as used for instance in the preparation of alcohols from ketones, in which similar types of catalysts can often be used.

A large number of catalysts are suitable for use in the production of ketones by the continuous catalytic dehydrogenation of secondary alcohols, including a wide variety of metals, metal oxides and mixtures thereof. For example ketones such as acetone, methyl ethyl ketone and diethyl ketone may be prepared from the corresponding secondary alcohols by dehydrogenation in the presence, as catalyst, of a metal such as copper or zinc or an alloy thereof such as brass. Oxides of many metals or mixtures of metals and oxides of the same or different metals, as for example oxides or mixtures of oxides supported on copper, zinc or brass are also suitable. If desired the oxide catalysts can be supported on inert materials such as

pumice, majolica or porcelain which take no significant part in the reaction and act merely as mechanical supports for the catalytically active material. Alloys of copper and zinc may advantageously be used, particularly those which have been pickled in mineral acid or for example acetic acid to produce a granular catalyst having a catalytically active outer layer. Many of these dehydrogenation catalysts have already been proposed for use in ketone production, and many proposals have also been made for improving the initial activity of such catalysts.

The preparation of ketones by catalytic dehydrogenation of secondary alcohols may be carried out by passing the appropriate secondary alcohol in vapour form through a reactor comprising for example a bank of tubes containing the dehydrogenation catalyst, the tubes being heated to a reaction temperature lying between about 300—500° C. thus the temperature may be between 380 and 420° C. initially, rising to say 480 to 500° C. as the reaction proceeds. The reaction may be effected under atmospheric or superatmospheric pressures e.g. up to about 5—10 atmospheres. Such tubular reactors may take a variety of forms, and may include the case in which some of the tubes contain no catalyst and serve primarily to vaporise the liquid alcoholic feed. It will be appreciated that other forms of reactor may also be used, as for example a single reactor containing either a fixed or a fluidised bed of catalyst.

Although the process is carried out in the vapour phase it is conveniently started up by passing the liquid feed through the whole of the reactor which is gradually heated to reaction temperature. In the case, for example, of a tubular reactor of the kind referred to above, which is heated by hot gases, the last row of tubes will be the first

to reach working temperature and consequently vaporisation of the alcoholic feed will occur in those tubes first. As the plant heats up the liquid/vapour interface moves back through the tubes until, under reaction conditions, vaporisation will be taking place in the first row of tubes and vapour phase ketone conversion will be occurring in all the catalyst-containing tubes, though mainly in the later row or rows of tubes. It will be appreciated that ketone production occurs gradually as warm-up proceeds and as the catalyst reaches working temperatures. This warm-up period, which includes the initial condition in which the liquid feed contacts substantially the whole of the catalyst, will hereinafter be referred to as the "initial stages" of the process.

As in the case of many catalytic reactions carried out on a technical scale, the activity of the dehydrogenation catalyst used in aliphatic ketone production tends to decline during the course of the process and it is usual in practice gradually to raise the reaction temperature as the reaction proceeds in order to compensate for this decline in activity. There is however a limit to the extent to which the reaction temperature can be raised and consequently it has hitherto been necessary to stop the reaction when the maximum working temperature has been reached and to remove the catalyst and either to regenerate it or to replace it with fresh catalyst altogether. Thus for example in processes for the preparation of acetone from isopropyl alcohol using a brass spelter catalyst it has been found necessary to remove the catalyst at intervals and to pickle it in mineral acid in order to remove surface impurities and to expose a fresh catalytically active surface. It is particularly desirable to improve the activity of the catalyst so as to increase the conversion obtainable therewith and since the removal and regeneration of the catalyst also adds to the cost of ketone production it is also desirable to reduce this expenditure as far as possible by improving the nature of the catalyst and/or the regeneration treatment so as to prolong the period before regeneration is required.

It has now been found possible to activate or otherwise to improve the nature of catalysts such for example as the dehydrogenation catalysts used in ketone production and to effect such improvement or activation of the catalyst *in situ* in the reactor by treating the catalyst during the initial stages of the process with a compound or a plurality of compounds which may be a salt or salts (advantageously an organic acid salt or salts) of the catalyst metal (or of one or more of the catalyst metals if more than one catalyst metal is present in the catalyst) or even of another metal or metals having dehydrogena-

tion activity, provided the compound or compounds is or are such as will decompose subsequently to deposit a catalytically active compound of the metal or metals, which is usually an oxide thereof, on the catalyst, thereby enhancing the activity of said catalyst in use.

According to the present invention catalyst activation is effected in a continuous vapour phase catalytic process for the preparation of an oxygen-containing organic compound from a liquid starting material which contacts substantially the whole of the catalyst in the initial stages of the process by continuously or intermittently adding to the liquid starting material supplied during said initial stages a metal compound or metal compounds, which compound or compounds will decompose to form a catalytically active deposit on the catalyst.

According to a particular application of the present invention catalyst activation is effected in a continuous catalytic dehydrogenation process for the preparation of a ketone from a secondary alcohol by adding to the liquid alcoholic feed supplied in the initial stages of the process a compound of a metal having dehydrogenation activity or a plurality of such compounds, which compound or compounds will be deposited on the catalyst and will subsequently decompose during the initial stages of the process to form a catalytically active deposit on the catalyst, the addition of said compound or compounds being made either intermittently or continuously to said liquid alcoholic feed during the initial stages of the process.

The catalyst may be treated in the manner just defined at intervals during the process for example at periodic intervals of between 500 and 1000 hours. A significant improvement in catalyst activity is thereby obtained, the catalyst being activated *in situ* in the reactor in such a manner that there is no possibility of the metal compound or compounds deposited on the surface of the catalyst being removed by the liquid starting material during the initial stages of the process. Also there is no danger of the catalytically active material deposited on the surface of the catalyst being removed accidentally in the handling and charging operations which would otherwise be required if the catalyst were activated in a separate operation effected outside the reactor, quite apart from the advantage that activation *in situ* is more convenient and less costly than activation in such a separate operation. Moreover, it has been found that a greater amount of catalytically active material can be deposited on the catalyst when the treatment is carried out *in situ* in the reactor in accordance with the present invention and that as a result of the treatment the activity of the catalyst—as repre-

sented by the degree of conversion which can be achieved therewith—is significantly enhanced.

It is also within the scope of the present invention to effect catalyst regeneration in a catalytic process as broadly defined above by interrupting the process, allowing the plant to cool down to below the vaporisation temperature of the liquid feed, and thereafter treating the catalyst with said metal compound or compounds in the manner defined above during the initial stages of the subsequent run.

The total amount of said compound or compounds added, either in the initial activation of the catalyst or in a subsequent regeneration treatment (e.g. in the manner just defined), may be between 1 and 10, e.g. about 6 to 7 pounds per ton of catalyst.

When a metal salt or a plurality of metal salts is or are employed it is convenient to add the salt or salts in aqueous solution, although addition in an alcohol-miscible non-aqueous medium is also possible providing appropriate metal compounds are used.

The process of the present invention is particularly suitable for the production of the lower aliphatic ketones from secondary alcohols containing up to about 4 carbon atoms in the molecule. The alcohol feed may be substantially anhydrous or it may be in admixture with water, e.g. as an azeotrope or constant boiling mixture. Such constant boiling mixtures are usually formed in the industrial preparation of the alcohols by hydration of the corresponding olefins and consequently are conveniently utilised as feedstocks for ketone production.

In general the dehydrogenation catalysts which can be used in the preparation of aliphatic ketones from aliphatic secondary alcohols in accordance with the present invention comprise one or more metals such as copper, silver, chromium, manganese, nickel, tungsten, cobalt, iron, cadmium, uranium, tin and zinc, either as such or in the form of their oxides or hydroxides (which may be formed by decomposition of the metal salts with organic or inorganic acids) and such catalysts may be used as such or supported on inert materials such as kieselguhr, pumice or majolica. The use of support materials such as activated alumina having dehydrating activity should be avoided otherwise dehydration of the alcohol feed occurs to give appreciable quantities of olefins as an unwanted by-product. A particularly suitable support for a metal oxide catalyst such as copper oxide is porcelain.

Whilst the present invention in its broadest aspect is not concerned with the particular catalyst employed in the dehydrogenation reaction or the method of preparing the catalyst, it has been found to be particularly

advantageous when the catalyst comprises or includes the metals copper and/or zinc or an oxide or oxides thereof, which oxides may be supported on the metals or on inert supports such as porcelain e.g. in the form of beads or rings. An alloy of copper and zinc such as brass, e.g. in the form of brass spelter which has been pickled in acid to expose a catalytically active surface, may also be used as the catalyst and/or support.

Thus according to a preferred feature of the present invention catalyst activation in a continuous process for preparing an aliphatic ketone from the corresponding secondary alcohol by dehydrogenation thereof in the presence of a dehydrogenation catalyst which comprises or includes the metals copper and/or zinc or an oxide or oxides thereof, is effected by continuously or intermittently adding a zinc salt and/or a copper salt to the liquid alcoholic feed supplied in the initial stages of the process.

The salt is preferably an organic acid salt and for example zinc acetate has been found to be particularly effective for activating a brass spelter catalyst used for example in the production of acetone from isopropyl alcohol and which has been pickled in mineral acid prior to being charged to the reactor. However, other organic acid salts of zinc and/or copper may be used, the salts being deposited on the catalyst during the initial stages of the process and thereafter decomposing to give the oxide.

The process of the present invention may be illustrated by the following Example:—

EXAMPLE.

Acetone was prepared from isopropanol using the isopropanol/water constant boiling mixture as the feedstock. This feedstock was passed in the vapour phase over a brass spelter catalyst in a tubular type reactor having narrow tubes of less than 6 inches diameter, the initial stages of the process being effected as set forth above.

The catalyst, which had been previously pickled in a mineral acid bath, was maintained at about 400° C. during the early part of the process, the temperature being raised gradually to a maximum of about 490–500° C. as the reaction proceeded.

Comparative runs were carried out (a) without any *in situ* catalyst treatment and (b) with the following treatment of the catalyst:—during the initial stages of the process 0.5 gallon of 10% (by weight) aqueous zinc acetate was added to the liquid feed at hourly intervals for a period of 8 hours.

Run (b) continued for over 1000 hours with not less than a 70 mol per cent conversion. In run (a), i.e. without *in situ* catalyst treatment, the process continued for only about

700 hours before the conversion fell below 70 mol per cent.

As indicated previously many of the catalysts referred to above as dehydrogenation catalysts can also function as catalysts in other reactions such as hydrogenation reactions; for example a zinc or brass spelter catalyst can be used in the production of isopropyl alcohol by hydrogenation of acetone and it will be understood that the

catalyst activation treatment of the present invention may also be used in such cases.

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